(19) Japanese Patent Office (JP)
(12) Publication of Unexamined Patent Application (A)

(11) Unexamined Patent Application No.:	S63-26373
(43) Unexamined Patent Application Date:	February 3, 1988
Request for Examination:	Not yet submitted
Number of Inventions:	1
Total pages:	3

(51) Int.CL[4]	Identification Symbol	JPO File Number	FI	Technology Display Area
C 23 C 16/50		6554-4K		
16/34		6554-4K		

(54) Title of Invention: A Method for Coating the Inside Surface of a Tube with Plasma CVD

(21) Patent Application No.: S61-169185

(22) Patent Application Date: July 18, 1986

(72) Inventor: Yasuyuki Yamada
4-8-1 Tsutsujigaoka, Tarumi-ku, Kobe, Hyogo Prefecture

(71) Applicant: Kobe Steel, Ltd.
3-18 Wakihama-cho 1-chome, Chuo-ku, Kobe, Hyogo Prefecture

(74) Agent: Hisashi Nakamura, Patent Attorney

EXHIBIT 1
S.N. 09/529,192
Office Action
dated 5-12-03

Specification

1. Title of the Invention

A Method for Coating the Inside Surface of a Tube with Plasma CVD

2. Claims

With regard to a method for coating the inside surface of a tube with plasma CVD, the electrically conductive tube to be coated is used as the first electrode and a gas nozzle having outlets in the longitudinal direction is installed in the first electrode tube to introduce reaction gas into the tube. After forming a vacuum, reaction gas is introduced into the first electrode. This method of coating the inside surface of a tube with plasma CVD is characterized by coating the inside surface of the tube by generating hollow-cathode discharge in the tube by applying high voltage between the first electrode and a vacuum chamber, which acts as the second electrode.

2. Detailed Explanation of the Invention

(Industrial Field of Application)

This invention relates to a technology for coating the inside surface of a tube. In particular, it relates to a method for coating the inside surface of a long tube using the plasma CVD method.

(Prior Art and Problems the Invention is to Solve)

The ion plating method and various other methods have been developed for coating the inside surface of a tube.

When the plasma CVD method is used, the inside surface of the tube is coated by placing the coating material in the tube to be coated and generating glow discharge in the tube by introducing electrons from the outside (e.g., Laid-Open Patent S53-1139, 53-1140). Alternatively, the inside surface of the tube is evenly coated by installing a movable plasma generating device or an evaporating source and moving it in the axial direction of the tube (Laid-Open Patent S49-36543, 53-31581).

However, with the former method, it is difficult to form a homogeneous coating inside the tube. And, with the latter method, an evaporation source must be installed inside the tube, consequently, the device becomes complex. In particular, it is difficult to coat the inside surface of a long tube having a small diameter. With either method, it is difficult to coat the inside of the tube evenly.

The objective of this invention is to solve the problem described above and to provide a method for

evenly coating the inside surface of a tube with a simple configuration, in particular, the inside surface of a long tube, a tube with a large diameter, a tube with a small diameter, and a long tube [*sic*].

(Means of Solving the Problem)

In order to achieve the objective described above, the inventor did research on a simple method that would not require the insertion of a complex device inside the tube to be coated. In addition, the inventor discovered that the electron concentration increases and, hence, the ionization ratio of the reaction gas will also increase when the tube is used as an electrode, when an immovable gas nozzle is installed inside the tube, and when hollow-cathode discharge is generated inside the tube.

According to the method for coating the inside surface of a tube with plasma CVD in this invention, an electrically conductive tube to be coated is used as the first electrode, and a gas nozzle having outlets in the longitudinal direction is installed in the first electrode tube to introduce reaction gas into the tube. After forming a vacuum, reaction gas is introduced into the first electrode. This method of coating the inside surface of a tube with plasma CVD is characterized by coating the inside surface of the tube by generating hollow-cathode discharge in the tube by applying high voltage between the first electrode and a vacuum chamber, which acts as the second electrode.

This invention is explained in detail below based on the working example.

Figure 1 shows one example of the plasma CVD device used in the working example of this invention.

In this figure, 1 is a vacuum chamber having an exhaust port, 2. A heater, 3, is installed inside the chamber. A fixed or rotate-able support, 4, penetrates one end of chamber 1 via an insulator and is airtight. A passage, 5, for introduction of reaction gas is formed through support 4. 10 is a valve for switching this passage on and off. Support 4 and vacuum chamber 1 are wired via a bias DC power supply, 6.

The tube, 7, to be coated is placed on the support, which protrudes into the chamber. A tubular gas nozzle, 8, is located inside the tube. Gas nozzle 8 has many reaction gas outlets, 9, along the wall. Outlets 9 are arranged at uneven intervals in the longitudinal direction of the nozzle. Typically, outlets 9 are arranged so that the concentration of the reaction gas becomes uniform inside the tube over the entire length, and the intervals become smaller as the distance from the reaction gas entrance increases. The inside diameters of outlets 9 can be determined as appropriate; the inside diameters do not need to

be equal. It is recommended that the end of gas nozzle 8 be closed.

A horizontally placed vacuum chamber, multiple supports, and numerous other variations are also possible.

(Working Example)

The inside surface of an SUS tube ($\phi 10 \times 200\ l$) is coated using the plasma CVD device shown in Fig. 1.

After forming a vacuum of 1×10^{-4} Toor [*sic*] or less inside the vacuum chamber, the tube is heated to 350 ~ 600 °C, preferably to 450 °C, with a heater and held for 20 ~ 30 minutes.

Next, mixed gas of $TiCl_4$, H_2, N_2, and Ar is introduced into the tube while the heater is still on. The pressure is maintained at 0.5 ~ 10 Torr. Simultaneously, -300 ~ -450 V bias is applied to the tube. Then very bright glow discharge, i.e., hollow-cathode discharge, is generated inside the tube.

After the set time period, all switches are turned off and the coating is completed.

This tube was examined after the process. A TiN coating was formed along the inside surface as well as on the outside. The coating thickness distribution was very uniform.

A straight tube was used in the working example described above but this method can also be applied for coating a curved tube or tubes with other shapes.

(Effect(s) of the Invention)

As described above, the plasma CVD method of this invention can uniformly coat the inside surface of tubes having various sizes and shapes. Because the device is simple, this method is practical and contributes to cost cutting.

4. Brief Explanation of the Drawings

Figure 1 is a cross-section of the plasma CVD device used in the working example of this invention.

1 ··· Vacuum chamber, 2 ··· Exhaust port, 3 ··· Heater, 4 ··· Support, 5 ··· Reaction gas passage, 6 ··· Bias DC power supply, 7 ··· Tube to be coated (First electrode), 8 ··· Gas nozzle, 9 ··· Gas outlet, 10 ··· Valve.

Patent Applicant: Kobe Steel, Ltd.

Patent Attorney: Hisashi Nakamura

EXHIBIT 1
S.N. 09/529,192
Office Action
dated 5-12-03

Figures

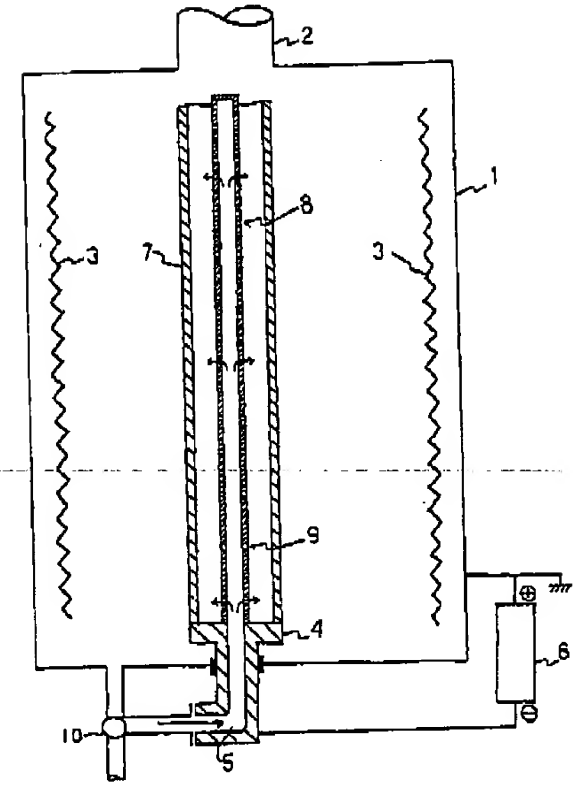

Figure 1

EXHIBIT 1
S.N. 09/529,192
Office Action dated 5-12-03

PHONE (734) 973-6101
FAX (734) 973-1847
jamltdmi@ameritech.net

JAPAN-AMERICA MANAGEMENT, LTD.

- ENGINEERING & MANAGEMENT CONSULTING
- TRANSLATION & INTERPRETING

2020 HOGBACK RD., SUITE 17
ANN ARBOR, MICHIGAN 48105

Certification

Document translated: **JP Publication No. S63-26373**

This is to certify that the above-stated document was translated by Japan-America Management, Ltd. from Japanese into English, and that it represents an accurate and faithful rendition of the original text to the best of my knowledge and belief.

By:

Mario Ciricola
Manager
April 28, 2003

EXHIBIT 1
S.N. 09/529,192
Office Action
dated 5-12-03